

**National Exposure Research Laboratory
Research Abstract**

Government Performance Results Act (GPRA) Goal 5
Annual Performance Measure 64

Significant Research Findings:

**Guidance to Identify, Quantify, and Reduce Laboratory Subsampling
Errors in Soils and Solid Wastes****Scientific
Problem and
Policy Issues**

Nearly all environmental research programs require the collection of samples in the field. However, the overwhelming majority of efforts (in terms of time and cost) to control and quantify error components in the data are concentrated on laboratory analyses. It has been repeatedly stated that 80% of the total error occurs in the field for the more stable contaminants (e.g., metals, PCBs, and pesticides) and up to 99.99% of the total error occurs in the field for non-stable contaminants (e.g., volatile organic compounds; (VOCs)). This research effort is aimed at examining and reducing the biases (intentional or unintentional error) that occur due to the use of “improper” sampling techniques when collecting the analytical subsample. An improperly collected analytical subsample will, in turn, yield contaminant concentrations that are not representative of the site and can lead to improper decisions being made by regulators on whether or not to remediate a site.

A field of sampling theory and practices has been developed by Dr. Pierre Gy in which logical steps have been identified that can reduce the error associated with sample collection. The original sampling theory and practices were developed for the mining industry. However, the same errors can occur when sampling contaminated soils or other solid matrices. While the sampling theory and practices appear to be completely logical and the examples given indicate the sampling errors do occur, little testing has been done on environmental samples to determine if by following the guidelines and “rules” established by Dr. Gy, better (i.e., more accurate, precise, and representative) samples will be obtained in the environmental field.

If the Gy sampling theory and practices are valid in the environmental field, resultant data being given to decision makers and regulators will be more accurate, precise, representative of the site being sampled, and will be legally-defensible. With this improved data, decision makers can better define the extent and degree of contamination at any site. Further, with the more accurate and precise data, the need for remedial actions can be better determined and the health of the public can be better protected.

**Research
Approach**

The focus of this research effort was to examine sample reduction techniques that are typically used to take the large mass/volume field samples (typically ranging from hundreds of grams to kilograms) and to decrease their size down to a method defined analytical sample size (typically ranging from milligrams to hundreds of grams) for their effects on sampling error (i.e., either reducing or increasing sampling error).

To test the effectiveness of the selected sample reduction techniques, artificially-created, known contaminant distributions were created in the laboratory. Contaminants were represented by using magnetite and salt (i.e., sodium chloride) and the “soil” was represented by a pure sand. Artificially-created contaminant distributions included: even layering of contaminant and non-contaminant layers, using pockets of large particle sizes, using pockets of fine particle sizes, and using coated particles (i.e., salt-coated sand grains). Analytical results yielded either weights of collected magnetite or total salt concentrations.

Sample reduction techniques tested were sectorial splitters, riffle splitters, manual incremental sampling, grab sampling, and cone and quartering techniques. Sample grinding was also examined to determine if particle size reduction was successful in reducing error associated with sample collection. The sample reduction technique(s) that yielded results that had the least error were those that were most precise and the least biased (i.e., most accurate) based on the known contaminant distribution and concentrations. To ensure the integrity of the contaminant distributions, an exhaustive analysis (analysis of the entire sample distribution) was performed and favorably compared against the known amounts of contaminants placed in the sample.

**Results and
Impact**

The laboratory sample reduction techniques, in general, reflected agreement of Gy sample theory and practices, indicating that these techniques and practices can be used successfully in the laboratory to reduce the error associated with the collection of environmental samples.

The best sample reduction technique was the sectorial splitter. This technique was fast, easy to use, and essentially eliminated all error that is typically associated with poor sample collection (i.e., these samples gave the minimum error expected). The use of riffle splitters to reduce sample size gave comparable results to the sectorial splitter. Manual incremental sampling, a process in which about 30 small samples are collected and combined together to represent the final analytical samples, was cumbersome and tedious to perform and yielded greater variability in sample results (i.e., greater error that can lead to incorrect assessments of the contaminant concentrations at a site) than either the sectorial or riffle splitters. Cone and quartering techniques and grab sampling failed to produce representative results as indicated by higher variability among the replicated samples. For example, the error associated with grab sampling was over an order of magnitude greater than the error measured by the use of the sectorial splitter. These two methods also tended to take longer to produce the final analytical sample. Sample grinding is highly recommended for all samples,

either before or after employing the sample reduction techniques and where practical, since this technique yielded the most precise and accurate contaminant concentrations when compared to the known values.

This research project helps satisfy the FY02 Annual Performance Goal (APG) 9 entitled, “Provide at least 2 new soil sampling and on-site screening methods.” This APG is part of the larger Government Performance Results Act (GPRA) subobjective that is aimed at improving site characterization, site monitoring, and modeling of contaminant fate and transport in the environment. In brief, following Gy’s sampling theory and practices, a significant reduction in sampling error can be achieved with little additional effort on the part of the sample collector or the analytical laboratory. And finally, by knowing with greater accuracy and precision what contaminants are present, where they are located, and what will happen to them if they remain untreated at a site, regulators and the general public can more appropriately prescribe actions to protect human health and the environment.

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| Research Collaboration and Research Products | <p>This research project was conducted primarily by a team of National Exposure Research Laboratory (NERL) staff scientists and staff scientists at the National Enforcement Investigation Center (NEIC) in Golden, Colorado. Contractor support was obtained from EnviroStat of Fort Collins, Colorado, and Lockheed-Martin Environmental Services of Las Vegas, Nevada.</p> <p>One part of this research has been published in the following manuscript:</p> <p>Gerlach, R.W., D.E. Dobb, G.A. Raab, and J.M. Nocerino. Gy sampling theory in environmental studies. 1. Assessing soil splitting protocols. <i>J. Chemometrics</i>. 16:321-328, 2002.</p> |
| Future Research | <p>Research investigating the sampling error associated with commonly used sampling tools (e.g., scoops, shovels, coring devices) is planned in an effort to further reduce sampling error. Additionally, this research will be extended to test the principles and results obtained in the laboratory setting to the field setting where greater volumes of samples are collected and processed.</p> |

**Contacts for
Additional
Information**

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